10/553862

JC20 Rec'd PCT/PTO 21 OCT 2005

METHOD FOR PRODUCING HIGH-STRENGTH SUPERPLASTIC MATERIAL

Technical Field

[0001] The present invention relates to the grain refinement of a metal material based on the use of an ultrasonic wave and a method for producing a metal material having high-strength and superplastic characteristics.

Background Art

[0002] It is known that, the smaller the grain of a metal material, the higher the strength, toughness and corrosion resistance of the metal material. When the grain size of a metal material is not more than several micrometers, the superplasticity phenomenon occurs and workability is dramatically improved under specific heating conditions although exceedingly high strength is exhibited at room temperature.

[0003] According to a general definition of superplasticity, superplasticity is considered to be a phenomenon that in the stretch deformation of a polycrystalline material, deformation stress shows high strain dependence and a gigantic elongation of not less than several hundred percent is exhibited without the occurrence of local shrinkage. Concretely, it is said that a material having grains which are equiaxed and as small as not more than 10 μm, exhibits a gigantic elongation at a deformation stress of not more than 10 MPa when deformed at a temperature of not less than 1/2 of the melting point represented by absolute temperature and at a strain rate of 10-4/s or so.

[0004] As grain refining methods of ferrous materials and nonferrous metal materials, there are known methods which involve adding an element which suppresses the growth of grains, methods which involve using transformation,

precipitation and recrystallization by thermo-mechanical treatment, methods which involve applying heavy shear working, etc. (refer to, for example, JP2003-041331A, JP2002-194472A, JP2002-105568A, and JP2000-271693A).

[0005] For ferrous materials, methods which involve using transformation, precipitation and recrystallization by thermo-mechanical treatment are effective and fine-grained structures of less than 1 μm have been obtained on a laboratory scale. However, to what extent processes can be simplified to adapt to mass production is a problem.

[0006] On the other hand, for nonferrous metal materials and, in particular, aluminum, it has hitherto been difficult to obtain uniform fine-grained structures of not more than 10 μm. In Japan, as a project for making fine-grained structures of not more than 3 μm by the New Energy Development Organization (NEDO), technology development has been carried out in a five-year plan since 1997. Fundamental techniques of the project are methods which involve applying heavy shear working to materials.

[0007] In recent years, light-weight and tough magnesium alloys having high vibration absorption characteristics have been used in the cases of a notebook personal computer and a cellular phone. However, magnesium the crystal structure of which is a hexagonal close-packed structure has low elongatability at room temperature and secondary working such as pressing is difficult. Thus, magnesium has the disadvantage that good quality parts and cases cannot be obtained unless shaping is performed by die casting and thixomolding. Such limitations to manufacturing methods make the applications of magnesium alloys narrow.

[0008] Furthermore, insufficient strength of magnesium alloys is part of the reason why the application of magnesium alloys to transportation equipment such as automobiles and aircraft does not advance.

[0009] In order to solve this problem, technology development for obtaining fine-grained grains of not more than 1 μm is being examined. One of such

techniques is methods which involve applying heavy shear working as with aluminum.

[0010] Although in metal materials, extrusion and rolling by rolls are general methods of applying heavy shear working, recent years have seen studies on the ECAP process (the equal-channel-angular pressing process) etc.

[0011] In extrusion, a billet or a slab is extruded from a die having an opening of a specified shape and the direct method by which a billet or a slab is extruded through an orifice of a die is generally adopted. For example, in the case of pure magnesium, a billet or a slab which is heated from 350 to 400°C is extruded. However, compared to aluminum, the balance between billet temperature and extrusion speed is difficult, and pure magnesium has the disadvantage that the material is not extruded when the temperature is low even a little and oxidation occurs when the temperature is raised. In the case of an Mg-Al-Zn alloy (AZ-Alloy) etc., further precise control is necessary.

[0012] Rolling by rolls is a method by which a metal material is discharged in one direction while being pressurized by top and bottom rolls, and accumulative roll bonding, cryogenic rolling, rolling with differential peripheral speeds, molten metal rolling, warm rolling, etc. are being studied.

[0013] In accumulative roll bonding, a rolled plate is divided into two in the direction of the length and subjected to surface treatment such as degreasing, and after that, the two plates are superposed on each other and rolled again. Although this method is characterized in that heavy shear working is possible without changing plate thickness, it has the disadvantage that production cost is high.

[0014] Cryogenic rolling is a method by which rolling is performed at a liquid nitrogen temperature at which strains introduced in rolling are not restored as far as possible, and after that, the formation of fine recrystallized grains is aimed at by rapid heating. However, sufficient effects have not been obtained.

[0015] Rolling with differential peripheral speeds is a method by which heavy shear working is applied to a material by changing the peripheral speed of top and bottom rolls. Because rolling is performed without lubrication, the material is apt to be subjected to a nonuniform shearing force and this method has the disadvantage that the material surface is roughened.

[0016] Molten metal rolling is a method by which a molten metal, in which an added element has been dissolved in a solid solution state in a supersaturated manner, is rapidly cooled by being poured into water-cooled rolls or by other means. Although the added element is effective in promoting the formation of a crystallization nucleus and simultaneously suppressing grain growth, metal materials which are apt to be oxidized require a thorough atmosphere adjustment. Therefore, this method is not suitable for mass production.

[0017] Warm rolling is a method by which rolling is performed at temperatures intermediate between temperatures of hot rolling which are not less than the recrystallization temperature and room temperature of cold rolling. For example, in an alloy obtained by adding an appropriate amount of Zr to an Al-Zn-Mg-Cu alloy, fine-grained structures are obtained. Thus, the effect of this rolling method has been ascertained in some alloys. However, the control of intermediate temperatures is very difficult and there are many unclear points as to whether effects are obtained in other metal materials.

[0018] The ECAP process is a method by which a billet or a slab is put in a die having a hole with a specific angle, pressurized and extruded thereby to apply a heavy shearing force to the billet or the slab. This method has been attracted attention because it is very effective as a method of obtaining fine-grained structures. However, because a billet or a slab which has received a heavy shearing force is very tough and hard, secondary working such as rolling is difficult. If hot rolling is performed in order to improve workability, grains grow and strength and toughness

which are sufficient at practical level and high ductility are not satisfied. This is the state of affairs.

[0019] Incidentally, as a method of making up for the disadvantage of the ECAP process, there has been proposed the continuous shear deformation process (the Conshearing Process) in which the ECAP process is made continuous (Saitou and other two persons, "PROPOSAL OF NOVEL CONTINUOUS HIGH STRATING PROCESS - DEVELOPMENT OF CONSHEARING PROCESS," Advanced Technology of Plasticity, Vol. III, Proceedings of the 6th International Conference of Technology of Plasticity, Sept. 19-24, 1999, pp. 2459-2464).

[0020] All of the methods relate to the heavy shear working of a produced billet etc., and shear working requires very large stresses or the initial shape of a metal material cannot be maintained.

Disclosure of the Invention

[0021] The present invention has been made to solve the above-described problems in prior art and has as its object the provision of a method for producing a high-strength superplastic material which enables a high-strength superplastic material having a metal structure formed from fine grains to be easily obtained.

[0022] In a method for producing a high-strength superplastic material of the present invention, the above-described problems are solved by applying a supersonic wave to a metal material and then subjecting the metal material to a heating treatment at a temperature obtained by multiplying a melting point of the metal material represented by absolute temperature by 0.35 to 0.6.

[0023] In many cases, when a vibration is given to a metal material, the vibration is attenuated in the course of time and eventually stops. There are two mechanisms of the attenuation of a vibration. One is called external friction and this is the mechanism that vibration energy is discharged from a vibrating metal material to outside via the air etc. The other is internal friction and this is the

mechanism that in the interior of a metal material, vibration energy is converted to heat, strain, etc. Internal friction is called also damping capacity.

[0024] Damping capacity is classified as the following four according to the difference in the conversion mechanism of vibration energy:

[0025] Damping capacity which is caused by viscous fluid or plastic fluid occurring at an interface between a mother phase and the second phase.

[0026] Damping capacity which is caused by an irreversible movement of a magnetic domain wall.

[0027] Damping capacity which is caused by the disengagement of a dislocation from a locking point due to an impurity atom.

[0028] Damping capacity which is caused by the movement of a transformation twin boundary as at a boundary between a mother phase and a martensite phase.

[0029] In metal materials having especially large damping capacity, part of the vibration energy is consumed as heat or accumulated as strain by any of the transformation mechanisms of the above classifications (1) to (4). In a metal material in which strains are accumulated, large strains equivalent to or more than strains in a case where shearing is mechanically applied are introduced. Therefore, it might be thought that if the metal material is subjected to a heating treatment at a temperature obtained by multiplying a melting point of the metal material represented by absolute temperature by 0.35 to 0.6, then in the process of energy release due to the reconfiguration of lattice defects or mutual coalescence and disappearance of lattice defects, the structure of the metal material changes to a recrystallized structure formed from equiaxed fine grains.

[0030] Metal materials having a large damping capacity generally refer to those having a specific damping capacity (SDC) of not less than 10% and are generically called high damping metal materials etc. In pure metals, Mg, Ni and Fe have a large specific damping capacity. In alloys, Mg alloys, Mn-Cu alloys,

Mn-Cu-Al alloys, Cu-Zn-Al alloys, Cu-Al-Ni alloys, Fe-Cr alloys (12Cr steels), Fe-Cr-Al alloys, Fe-Cr-Mo alloys, Co-Ni alloys, Fe-Cr-Al-Mn alloys, Ni-Ti alloys, Cu-Zn-Al alloys, Al-Zn alloys, intergranular corrosion-resistant 18-8 stainless steels, Fe-C-Si alloys (rolled cast irons obtained by rolling flaky graphite cast iron or spheroidal graphite cast iron), etc. have a large specific damping capacity and called high damping alloys, vibration-damping alloys, vibration-proof alloys, etc.

[0031] As given by the following equation, specific damping capacity is expressed by the vibration energy loss rate per cycle of a vibrating body:

[0032] $SDC(\%) = (\Delta W/W) \times 100$

[0033] where W is vibration energy and ΔW is the energy lost in one cycle.

[0034] Among high damping metal materials having a specific damping capacity of not less than 10%, Mg or Mg alloys are best suited for the application of this method. In Mg, which has the largest damping capacity of all metal materials and has a specific damping capacity of not less than 60%, vibration energy is easily accumulated as strain, and by performing heating treatment at an appropriate temperature, it is possible to obtain a recrystallized structure formed from fine grains. Mg has relatively small strength and corrosion resistance. However, in Mg alloys which are improved in this respect by adding Al, Zn, Zr, etc. though damping capacity is lower than in Mg, part of ultrasonic vibration energy is accumulated as strain and a recrystallized structure formed from fine grains is obtained by heating treatment as a result of the synergistic effect combined to the effect of added elements. Thus, it is possible that further high strength and superplasticity are compatible with each other.

[0035] For Mg alloys, Mg-Al alloys, Mg-Al-Zn alloys, Mg-Zr alloys, Mg—Zn-Zr alloys, Mg-Mg2 Ni alloys, Mg-RE-Zn alloys (RE means rare earth), Mg-Ag-RE alloys (RE means rare earth), Mg-Y-RE alloys (RE means rare earth), etc. are known as practical-use alloys. Because damping capacity decreases when the amount of added Al increases, the specific damping capacity of Mg-10%Al alloys

(Al100), Mg-9%Al-1%Zn alloys (AZ91), Mg-6%Al-3%Zn alloys (AZ63), etc. is less than 10% among Mg-Al alloys and Mg-Al-Zn alloys.

[0036] It might be thought that as described in the conversion mechanism of vibration energy (3) above, the ultrasonic vibration energy applied to Mg or Mg alloys is consumed by the disengagement of a dislocation from a locking point due to an impurity atom or in the generation of deformation twins.

[0037] A metal material to which an ultrasonic wave has been applied is recrystallized by being subjected to a heating treatment at a temperature obtained by multiplying a melting point of the metal material represented by absolute temperature by 0.35 to 0.6. At temperatures higher than a temperature obtained by multiplying the melting point represented by absolute temperature by 0.6, there are energy losses for suppressing the growth of recrystallized grains and control is difficult. At temperatures lower than a temperature obtained by multiplying the melting point represented by absolute temperature by 0.35, only strain recovery, which is the phenomenon that part of the strain in a metal material disappears, occurs and recrystallized grains are not formed.

[0038] A crystallization temperature is in effect a temperature at which a metal structure subjected to cold working is completely changed by a one-hour heat treatment into a structure having new recrystallized grains, and this temperature is a characteristic value which changes depending on the kind and purity of the metal material, the degree of internal strain, etc. However, there is a tendency that the crystallization temperature converses on a given temperature as internal strain increases. That is, it might be thought that in a metal material subjected to a large internal strain, grain growth is suppressed by performing control, with the above-described temperature range serving as a rough standard, with the result that it is easy to obtain a desired high-strength superplastic material.

Best Mode for Carrying Out the Invention

[0039] No special limit is provided for the shape of a metal material. It is possible to use, for example, solidified powder compacts, or plate materials, bar materials and pipes which are wrought materials or formed bodies obtained by the press forming to a desired shape. Solidified powder compacts are solidified compacts made by compression shearing of powder sinters or powders, wrought materials are objects obtained by pressing or extruding castings or metal materials solidified after melting to a desired shape.

[0040] As methods of applying an ultrasonic wave to a metal material, there is available, for example, a method by which a horn connected to an ultrasonic vibrator is brought into close contact with a metal material and an ultrasonic wave is applied for a given time. In order to ensure that vibrations are efficiently transmitted from the horn to the metal material, it is also possible to put a grease etc. to between the horn and the metal material. However, a safe grease which does not easily deteriorate or ignite must be used. For example, silicone grease can be used.

[0041] Furthermore, it is also possible to cause vibrations to be transmitted via an intermediate medium, for example, by a method by which a metal material is put in water or an organic solvent and vibrations emitted from a horn are caused to be transmitted from the water or the organic solvent, and methods other than those described above may be used so long as the methods ensure efficient and safe transmission.

[0042] For the frequency, output and application time of an ultrasonic wave, optimum values must be determined by giving due consideration to the melting point, specific damping capacity, size, etc. of a metal material. For example, in the case of an elongated material (20 mm X 50 mm X 1.25 mm) of an Mg-3%Al-1%Zn alloy (AZ31), which is an Mg alloy of a high damping alloy, an ultrasonic wave having a frequency of 19 kHz and an output of 200 W can be applied for 5 to 60 seconds by use of a titanium alloy horn having a diameter of 22 mm.

[0043] A metal material to which an ultrasonic wave has been applied is heated at the recrystallization temperature for 1 hour. For example, AZ31 the recrystallization temperature of which is expected to be 180 to 230°C, for example, is heated at 230°C in a vacuum for 1 hour. If not in a vacuum, it is preferred that AZ31 be heated in an argon atmosphere. If AZ31 is heated in nitrogen, hydrogen or oxygen, AZ31 forms compounds with these elements, worsening the surface properties and mechanical properties. Incidentally, heating in the air is allowed if an oxidation resistant metal material is used.

[0044] After the application of an ultrasonic wave, the recrystallized metal material keeps its initial shape and the grain size becomes 1/10 to 1/150 the grain size before the application of an ultrasonic wave. For example, for an elongated material of AZ31 (20 mm X 50 mm X 1.25 mm), the size of the material does not change and a crystal structure having grain sizes of 150 to 200 μm becomes an equiaxed crystal structure of 1 to 15 μm. Thus, it is possible to improve this AZ31 to an AZ31 material which has high strength and develops superplasticity.

[0045] According to a production method of a high-strength superplastic material of the present invention as described above, it is possible to obtain a high-strength superplastic material the internal structure of which is uniform and which is formed from fine-grained structure without changing the shape of metal material.

[Embodiment 1]

[0046] As a metal material, a test piece 20 mm X 50 mm X 1.25 mm was cut out from an elongated material made of industrial pure Al (JIS alloy number: 1100) by use of a cutter with a peripheral cutting edge, and the surface of the test piece was swiftly cleaned with ethanol.

[0047] An ultrasonic homogenizer was used as application means of ultrasonic wave, an appropriate amount of silicone grease was applied to an end surface of a titanium alloy horn 22 mm in diameter, and with the above-described test

piece of an elongated material made of industrial pure Al pushed against the end surface by use of a jack, ultrasonic wave vibrations of 19 kHz and 300 W were applied for 60 seconds. This operation was repeated three times.

[0048] The test piece of an elongated material made of industrial pure Al to which an ultrasonic wave had been applied was put in a vacuum heating furnace and subjected to a heating treatment for 1 hour with a degree of vacuum of 5 Pa and at a heating temperature of 468 K, i.e., a heating temperature/melting point = 0.50.

[0049] Deformation and a dimensional change in the test piece of an elongated material made of industrial pure Al due to the above-described treatment were scarcely observed.

[0050] The tensile strength of the elongated material made of industrial pure Al subjected to the heating treatment was 180 MPa, and when elongation to fracture was investigated at 473 K and at a strain rate of 10-4/s, it showed 150%. Thus, it became apparent that the superplasticity phenomenon had occurred.

[0051] Furthermore, when a test piece for structure observation 10 mm X 10 mm X 1.25 mm was cut out and a simple observation of polarized light was conducted under an optical microscope after etching with 0.5% aqua regia, the grain size was about 15 μm. This value was 1/10 of 150 μm which was the grain size before ultrasonic wave application.

[Embodiment 2]

[0052] As a metal material, a test piece 20 mm X 50 mm X 1.25 mm was cut out from a cold-rolled material made of industrial pure iron by use of a cutter with a peripheral cutting edge, and the surface of the test piece was swiftly cleaned with ethanol.

[0053] An ultrasonic homogenizer was used as application means of ultrasonic wave, an appropriate amount of silicone grease was applied to an end surface of a titanium alloy horn 22 mm in diameter and with the above-described test piece of a cold-rolled material made of industrial pure iron pushed against the end

surface by use of a jack, ultrasonic wave vibrations of 19 kHz and 300 W were applied for 60 seconds.

[0054] The test piece of a cold-rolled material made of industrial pure iron to which an ultrasonic wave had been applied was put in a vacuum heating furnace and subjected to a heating treatment for 1 hour with a degree of vacuum of 5 Pa and at a heating temperature of 923 K, i.e., a heating temperature/melting point = 0.51.

[0055] Deformation and a dimensional change in the test piece of a cold-rolled material made of industrial pure iron due to the above-described treatment were scarcely observed.

[0056] The tensile strength of the cold-rolled material made of industrial pure iron subjected to the heating treatment was 700 MPa, and when elongation to fracture was investigated at 923 K and at a strain rate of 10-3/s, it showed 200%. Thus, it became apparent that the superplasticity phenomenon had occurred.

[0057] Furthermore, when a test piece for structure observation 10 mm X 10 mm X 1.25 mm was cut out and a simple observation of polarized light was conducted under an optical microscope after etching with a 1% ethanol nitrate solution, the grain size was about 10 μm. This value was 1/15 of 150 μm which was the grain size before ultrasonic wave application.

[Embodiment 3]

[0058] As a metal material, a test piece 20 mm X 50 mm X 1.25 mm was cut out from an elongated material made of AZ31 by use of a cutter with a peripheral cutting edge, and the surface of the test piece was swiftly cleaned with ethanol.

[0059] An ultrasonic homogenizer was used as application means of ultrasonic wave, an appropriate amount of silicone grease was applied to an end surface of a titanium alloy horn 22 mm in diameter and with the above-described test piece of an elongated material made of AZ31 pushed against the end surface by use of a jack, ultrasonic wave vibrations of 19 kHz and 200 W were applied for 15 seconds.

[0060] The test piece of an elongated material made of AZ31 to which an ultrasonic wave had been applied was put in a vacuum heating furnace and subjected to a heating treatment for 1 hour with a degree of vacuum of 5 Pa and at a heating temperature of 503 K, i.e., a heating temperature/melting point = 0.54.

[0061] Deformation and a dimensional change in the test piece of an elongated material made of AZ31 due to the above-described treatment were scarcely observed.

[0062] The tensile strength of the elongated material made of AZ31 subjected to the heating treatment was 300 MPa, and when elongation to fracture was investigated at 503 K and at a strain rate of 10-2/s, it showed 100%. Thus, it became apparent that the superplasticity phenomenon had occurred.

[0063] Furthermore, when a test piece for structure observation 10 mm X 10 mm X 1.25 mm was cut out and a simple observation of polarized light was conducted under an optical microscope after etching with a 1% ethanol nitrate solution, the grain size was about 5 μm. This value was 1/30 of 150 μm which was the grain size before ultrasonic wave application.

[Embodiment 4]

[0064] A test piece of an elongated material made of AZ31 to which an ultrasonic wave had been applied was put in a vacuum heating furnace and subjected to a heating treatment for 1 hour with a degree of vacuum of 5 Pa and at a heating temperature of 463 K, i.e., a heating temperature/melting point = 0.50. In other respects, the same operation as in Embodiment 3 was performed.

[0065] The tensile strength of the elongated material made of AZ31 subjected to the heating treatment was 310 MPa, and when elongation to fracture was investigated at 503 K and at a strain rate of 10-2/s, it showed 130%. Thus, it became apparent that the superplasticity phenomenon had occurred.

[0066] Furthermore, when a test piece for structure observation 10 mm X 10 mm X 1.25 mm was cut out and a simple observation of polarized light was

conducted under an optical microscope after etching with a 1% ethanol nitrate solution, the grain size was about 3 μm. This value was 1/50 of 150 μm which was the grain size before ultrasonic wave application.

[Embodiment 5]

[0067] A test piece of an elongated material made of AZ31 to which an ultrasonic wave had been applied was put in a vacuum heating furnace and subjected to a heating treatment for 0.5 hour with a degree of vacuum of 5 Pa and at a heating temperature of 523 K, i.e., a heating temperature/melting point = 0.57. In other respects, the same operation as in Embodiment 3 was performed.

[0068] The tensile strength of the elongated material made of AZ31 subjected to the heating treatment was 300 MPa, and when elongation to fracture was investigated at 503 K and at a strain rate of 10-2/s, it showed 100%. Thus, it became apparent that the superplasticity phenomenon had occurred.

[0069] Furthermore, when a test piece for structure observation 10 mm X 10 mm X 1.25 mm was cut out and a simple observation of polarized light was conducted under an optical microscope after etching with a 1% ethanol nitrate solution, the grain size was about 5 μm. This value was 1/30 of 150 μm which was the grain size before ultrasonic wave application.

[Embodiment 6]

[0070] As a metal material, a test piece 20 mm X 50 mm X 1.25 mm was cut out from an elongated material made of AZ31 by use of a cutter with a peripheral cutting edge, and the surface of the test piece was swiftly cleaned with ethanol.

[0071] An ultrasonic homogenizer was used as application means of ultrasonic wave, a titanium alloy horn 22 mm in diameter was installed in such a manner that the distance of an end surface of the horn from the test piece of an elongated material made of AZ31 which was immersed in pure water became 2 cm, and ultrasonic wave vibrations of 19 kHz and 240 W were applied for 300 seconds.

[0072] The test piece of AZ31 to which an ultrasonic wave had been applied was put in a vacuum heating furnace and subjected to a heating treatment for 1 hour with a degree of vacuum of 5 Pa and at a heating temperature of 453 K, i.e., a heating temperature/melting point = 0.49.

[0073] Deformation and a dimensional change in the test piece made of AZ31 due to the above-described treatment were scarcely observed.

[0074] The tensile strength of the elongated material made of AZ31 subjected to the heating treatment was 375 MPa, and when elongation to fracture was investigated at 503 K and at a strain rate of 10-2/s, it showed 233%. Thus, it became apparent that the superplasticity phenomenon had occurred.

[0075] Furthermore, when a test piece for structure observation 10 mm X 10 mm X 1.25 mm was cut out and a simple observation of polarized light was conducted under an optical microscope after etching with a 1% ethanol nitrate solution, the grain size was about 1 μm. This value was 1/150 of 150 μm which was the grain size before ultrasonic wave application.

[Comparative Example 1]

[0076] As a metal material, a test piece 20 mm X 50 mm X 1.25 mm was cut out from an elongated material made of AZ31 by use of a cutter with a peripheral cutting edge, and the surface of the test piece was swiftly cleaned with ethanol.

[0077] An ultrasonic homogenizer was used as application means of ultrasonic wave, a titanium alloy horn 22 mm in diameter was installed in such a manner that the distance of an end surface of the horn from the test piece of an elongated material made of AZ31 which was immersed in pure water became 2 cm, and ultrasonic wave vibrations of 19 kHz and 240 W were applied for 300 seconds.

[0078] The test piece of AZ31 to which an ultrasonic wave had been applied was put in a vacuum heating furnace and subjected to a heating treatment for 1 hour with a degree of vacuum of 5 Pa and at a heating temperature of 303 K, i.e., a heating temperature/melting point = 0.33.

[0079] Deformation and a dimensional change in the test piece made of AZ31 due to the above-described treatment were scarcely observed.

[0080] The tensile strength of the elongated material made of AZ31 subjected to the heating treatment was 260 MPa, and when elongation to fracture was investigated at 503 K and at a strain rate of 10-2/s, it showed 50%. Thus, it became apparent that the superplasticity phenomenon did not occur.

[0081] Furthermore, when a test piece for structure observation 10 mm X 10 mm X 1.25 mm was cut out and a simple observation of polarized light was conducted under an optical microscope after etching with a 1% ethanol nitrate solution, the grain size was about 150 μm and no change from the grain size of 150 μm before ultrasonic wave application was observed.

[Comparative Example 2]

[0082] A test piece of an elongated material made of AZ31 to which an ultrasonic wave had been applied was put in a vacuum heating furnace and subjected to a heating treatment for 1 hour with a degree of vacuum of 5 Pa and at a heating temperature of 533 K, i.e., a heating temperature/melting point = 0.62. In other respects, the same operation as in Comparative Example 1 was performed.

[0083] Deformation and a dimensional change in the test piece of an elongated material made of AZ31 due to the above-described treatment were scarcely observed.

[0084] The tensile strength of the elongated material made of AZ31 subjected to the heating treatment was 280 MPa, and when elongation to fracture was investigated at 503 K and at a strain rate of 10-2/s, it showed 80%. Thus, it became apparent that the superplasticity phenomenon did not occur.

[0085] Furthermore, when a test piece for structure observation 10 mm X 10 mm X 1.25 mm was cut out and a simple observation of polarized light was conducted under an optical microscope after etching with a 1% ethanol nitrate

solution, the grain size was about 30 μm. This value was 1/5 of 150 μm which was the grain size before ultrasonic wave application.

Industrial Applicability

[0086] According to a production method of a high-strength superplastic material of the present invention, it is possible to give large internal strains to a metal material and it is possible to easily obtain a high-strength superplastic material which has a metal structure formed from fine grains.